NATIONAL BUREAU OF STANDARDS REPORT

8345

THERMAL CONDUCTIVITY OF A SPECIMEN OF
ELECTROFORMED NICKEL

by

T. W. Watson and H. E. Robinson

Report to

The International Nickel Company, Inc.
New York, N. Y.

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Thermal Conductivity of a Specimen of Electroformed Nickel

by

Thomas W. Watson and Henry E. Robinson

1. INTRODUCTION

This report presents results of thermal conductivity and electrical resistivity measurements in the temperature range −162° to 540° C of a sample of electroformed nickel (radially-deposited on a nickel wire) submitted by the International Nickel Company, Inc., 67 Wall Street, New York, N. Y.

2. SAMPLE

The sample submitted was a bar about one inch in diameter, which was machined to yield a test specimen having a uniform diameter of 2.54 cm and a length of 37.0 cm.

The chemical composition of the specimen is given in Table 1, as determined from a spectrochemical analysis made by the National Bureau of Standards Spectrochemistry Section.

3. TEST APPARATUS AND METHOD

The thermal conductivity of the sample was determined by means of a steady-state flow of heat longitudinally in the bar specimen, with measurements of the temperatures existing at the ends of six consecutive, approximately 3.51-cm, spans along the central length of the bar. Each determination required a pair of tests at moderately different temperature conditions, and yielded values of thermal conductivity at six different mean temperatures. [1] [a]

The test apparatus is shown schematically in Figure 1.

The specimen, a bar approximately 37 cm long and of uniform external dimensions over the metering length, was supported at the top (coolant) end concentrically within a stainless steel guard tube of 0.8-cm wall thickness, which in turn was held concentrically within a cylindrical outer container. The specimen was drilled at each end with a 1.35-cm hole 5.5 cm deep. An electrical heater was inserted and secured in the hole at the bottom (hot) end by a completely-enclosing

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[a] See references in 6. REFERENCES

metal cap (in lieu of the strap shown in Figure 1), and the supporting fixture at the top end provided a liquid-tight connection for circulating a coolant through the top drill-hole.

Temperatures along the specimen were indicated by seven thermocouples located symmetrically about the longitudinal center of the specimen, spaced approximately 3.51 cm apart, with one additional thermocouple near the bottom end of the specimen. Thermocouples were similarly located in almost exactly corresponding longitudinal positions on the guard tube.

The guard tube was equipped near its lower end with an external circumferential electric heater, as shown. The guard tube was cooled at the top by means of a copper-tube coil soldered circumferentially at a position corresponding in effect to that of the specimen coolant well. Coolant (liquid nitrogen at −196° C or water at 40° C) was pumped through the guard coil and specimen well in series connection, as shown.

The electrical heater for the specimen consisted of a porcelain cylinder 1.25 cm in diameter and 5.2 cm long, threaded longitudinally with 26-gage nichrome heater wire. Its resistance at 25° C was approximately 21 ohms. Current was brought to the heater through relatively large heater leads, to which separate potential leads were connected at the point where they entered the porcelain core. The heater was energized by an adjustable constant-voltage d-c source. Heater current and voltage drop measurements were made using standard resistors and the high-precision manual potentiometer used for thermocouple observations. The guard was heated with alternating current governed by a sensitive temperature controller actuated by the guard temperature at a selected position.

The thermocouples were made from calibrated chromel and alumel 26-gage wires, welded by gas-oxygen flame to form a butt joint about 0.042 cm in diameter.

The thermocouple junctions were pressed into transverse grooves 0.04 cm wide by 0.05 cm deep and 0.6 cm in length in the convex surface of the bar and tightly secured by peening the metal around the groove. The bare thermocouple leads were individually insulated electrically with high-temperature flexible sleeving, and led out into the powder insulation in the same transverse plane as the junction (one wire in each direction around the bar), forming a 2.5-cm circle. The wires were brought out through the powder insulation near the guard tube. The thermocouples in the guard tube were electrically welded to form a spherical junction about 0.10 cm in diameter. The junctions in the guard were inserted into radially-drilled holes 0.11 cm in diameter and 0.17 cm deep, and tightly secured by punch-pricking the metal around

the hole. The wires were similarly brought out through the powder insulation. The longitudinal positions of the thermocouple junctions were taken as those of the centers of the grooves, or of the drilled holes, measured to the nearest 0.01 cm with a laboratory cathetometer.

Current leads (0.1 cm Pt) were attached to the two ends of the bar specimen for passing a direct current of about 8 amperes along the bar for making electrical resistivity measurements. The lead at the hot end was led in a flat spiral in the powder insulation, in a plane transverse to the bar axis, to near the inner radius of the guard tube, from which point it was electrically insulated with broken ceramic tubing and brought upwards through the powder insulation near the guard tube.

After installation of the specimen, the space between it and the guard tube was filled with diatomaceous earth powder insulation, which also was used to insulate the space surrounding the guard tube. The tests were conducted with the insulation exposed to atmospheric air.

In principle, if there were no heat exchange between the specimen and its surroundings, the conductivity could be determined from the measured power input to the specimen and the average temperature gradient for each of the six spans along the specimen, all of uniform known cross-sectional area. In practice, a perfect balance of temperatures between the bar and guard all along their lengths is not possible because of differences in their temperature coefficients of conductivity, and the effect of the outward heat losses of the guard. In addition to heat exchanges between the bar and guard from this cause, a relatively smaller longitudinal flow of heat occurs in the powder insulation surrounding the specimen, and the contribution of the specimen to this heat flow must depend somewhat on the bar-to-guard temperature unbalance.

In order to evaluate the heat flow in the bar at the center points of each of the six spans, a partly empirical procedure was used. Two steady-state test-runs were made with slightly different bar and guard temperatures and power inputs. In the two tests, the heat flow and the observed temperature drop from end to end of a given span differed, as did also the approximate integral with respect to length of the observed temperature differences between bar and guard, summed from the hot end of the bar to the span center point. It is thus possible to write for each span two equations (one for each test-run) of the form

$$\frac{Ak\Delta t}{\Delta x} + fS = Q$$

where A is the cross-sectional area of the specimen,

k is the specimen conductivity at the mean temperature of the span,

Δt is the temperature drop from end to end of the span,

Δx is the length of the span,

fS represents the total net heat loss from the bar from its bottom end at the heater to the midpoint, x, of the given span, expressed as the product of S, which is the integral $\int_0^x (t_{bar} - t_{guard})dx$, and an average heat transfer coefficient f for the thermal path from bar to guard,

Q is the measured power input to the specimen heater.

The two equations written for each of the six spans of the bar can be solved simultaneously to determine k and f. For this to be strictly valid, k and f must have equal values in the two equations. Since the mean temperatures of the span in the two tests will in general differ slightly, and the conductivity of the bar may vary with temperature, a slight adjustment is made to the observed values of Δt so that k corresponds to the mean of the span mean temperatures in the two tests. The equality of f in the two tests is not so readily assured, but because the magnitude of fS in these tests was generally on the order of one percent of Q, a moderate difference in the values of f in the two equations would affect the solved value of $Ak/\Delta x$ only slightly.

Electrical resistivity measurements for each span were made at the end of, but at the temperature conditions existing at, each pair of runs for determining the thermal conductivity, by passing a d-c current of about 7.7 A along the bar, and observing the potentials of the chromel leads of the span thermocouples, with the current direction forward and reversed. The average of the two potential drops between two adjacent chromel leads indicated the net potential drop due to the current flowing in the span, and thus enabled calculation of its resistivity. Due to a slight warming of the bar during the period of current flow, the resistivity was assigned to correspond to the time-average of the span mean temperature over this period. The same electrical and temperature measurements also enabled calculation of the thermoelectric power of the nickel relative to the chromel P thermocouple wire used.

The computation of results directly from the observed data was effected by an IBM 7094 digital computer suitably programmed to compute the thermal conductivity, the electrical resistivity and the thermoelectric power, and the corresponding mean temperatures, for each of the six spans.

4. RESULTS

The results of the thermal conductivity and electrical resistivity determinations are shown in Figure 2 and Table 2. The 36 individual values plotted in each case represent six sets of tests, each with values for the six spans. The open circles represent values obtained in tests made with increased values of span mean temperatures; solid circles represent values obtained on cooling from the maximum temperatures attained. The solid lines represent the trend of the data from which the values tabulated in Table 2 were taken.

The calculated values of the thermoelectric power of the electroformed nickel relative to the chromel P thermocouple wire used are plotted in the upper part of Figure 3. Values of the Lorenz function, $k\rho/T$, were calculated from the values of thermal conductivity, electrical resistivity, and temperature tabulated in Table 2. They are given in Table 2 and represented by the smooth curve in the lower part of Figure 3.

5. DISCUSSION OF RESULTS

The individual values of thermal conductivity and electrical resistivity plotted in Figure 2 show moderate scattering from the smooth curve. The extreme departure of an individual value of thermal conductivity from the smooth curve is less than two percent. The uncertainty in the smoothed conductivity values is believed to be not more than one percent.

As shown in Figure 2, the thermal conductivity decreased as the temperature increased to the Curie point, and thereafter increased. The curves are shown dotted in the region where a part of a measuring span was at the apparent Curie temperature, because of increased uncertainty in the values obtained in this region. The dashed lines in Figure 2 represent the values of Van Dusen 1934 [2] for nickel of 99.94 percent purity.

The obtained values of electrical resistivity are shown in the upper part of Figure 2. The resistivity increased with temperature to the Curie point, and thereafter increased more slowly. The points plotted as triangles near the resistivity curve show values of resistivity reported by Roeser 1941 [3] for nickel of 99.94 percent purity; the squares show values of White and Woods 1959 [4] for nickel of "ideal purity."

The obtained values of thermoelectric power of the electroformed nickel, relative to the chromel P thermocouple wire used, are shown in Figure 3, with symbols consistent with those of Figure 2. The plotted triangles represent values of thermoelectric power of nickel relative to chromel P derived from data on thermal emf of nickel, and of chromel P, relative to platinum [5].

The lower curve of Figure 3 represents the values of the Lorenz function ($k\rho/T$) given in Table 2, derived from the thermal conductivity and electrical resistivity results. The theoretical (Sommerfeld) value 2.443×10^{-8} V^2/deg^2 is shown by the horizontal dashed line. In the neighborhood of the Curie point, the Lorenz function obtained using the conductivity and resistivity values given by the dashed parts of Figure 2 shows an upturn, for both parts of the curve in Figure 3. The upturns probably represent too-large values of k, or ρ, or both, obtained in the near region of the Curie point as a result of the considerable temperature difference from end to end of a measuring span in this region.

As inferred from intersections of the various curves from lower and higher temperatures, the approximate apparent values of the Curie temperature of the electroformed nickel specimen are 376° C from the thermal conductivity data, 376° C from the electrical resistivity data, and 378° C from the thermoelectric power data. These values compare with the value 358° C given for nickel by Bozorth [6].

6. REFERENCES

[1] T. W. Watson and H. E. Robinson, Thermal conductivity of some commercial iron-nickel alloys, ASME Jour. of Heat Transfer (November 1961) p. 403-408.

[2] M. S. Van Dusen and S. M. Shelton, Apparatus for measuring thermal conductivity of metals up to 600 C, J. Research NBS, 12, 429 (1934) RP668.

[3] W. F. Roeser, Temperature, Its Measurement and Control in Science and Industry, p. 1312 (Reinhold Publishing Corp., New York, N. Y., 1941).

[4] G. K. White and S. B. Woods, Electrical and thermal resistivity of the transition elements at low temperature, Phil. Trans. Roy. Soc. 251A, 273 (1959).

[5] American Institute of Physics Handbook, p. 4-8, 9 (McGraw-Hill Book Co., Inc., 1963, Second Edition).

[6] R. M. Bozorth, Ferromagnetism, p. 270 (Van Nostrand, 1951).

TABLE 1

Chemical Composition--Percent

Ni*	Co	Cu	Fe	Al
99.85	0.11	0.026	0.006	0.001

Si	Ti	Mg	Cr	Mn
< 0.004	< 0.002	< 0.001	< 0.001	< 0.0005

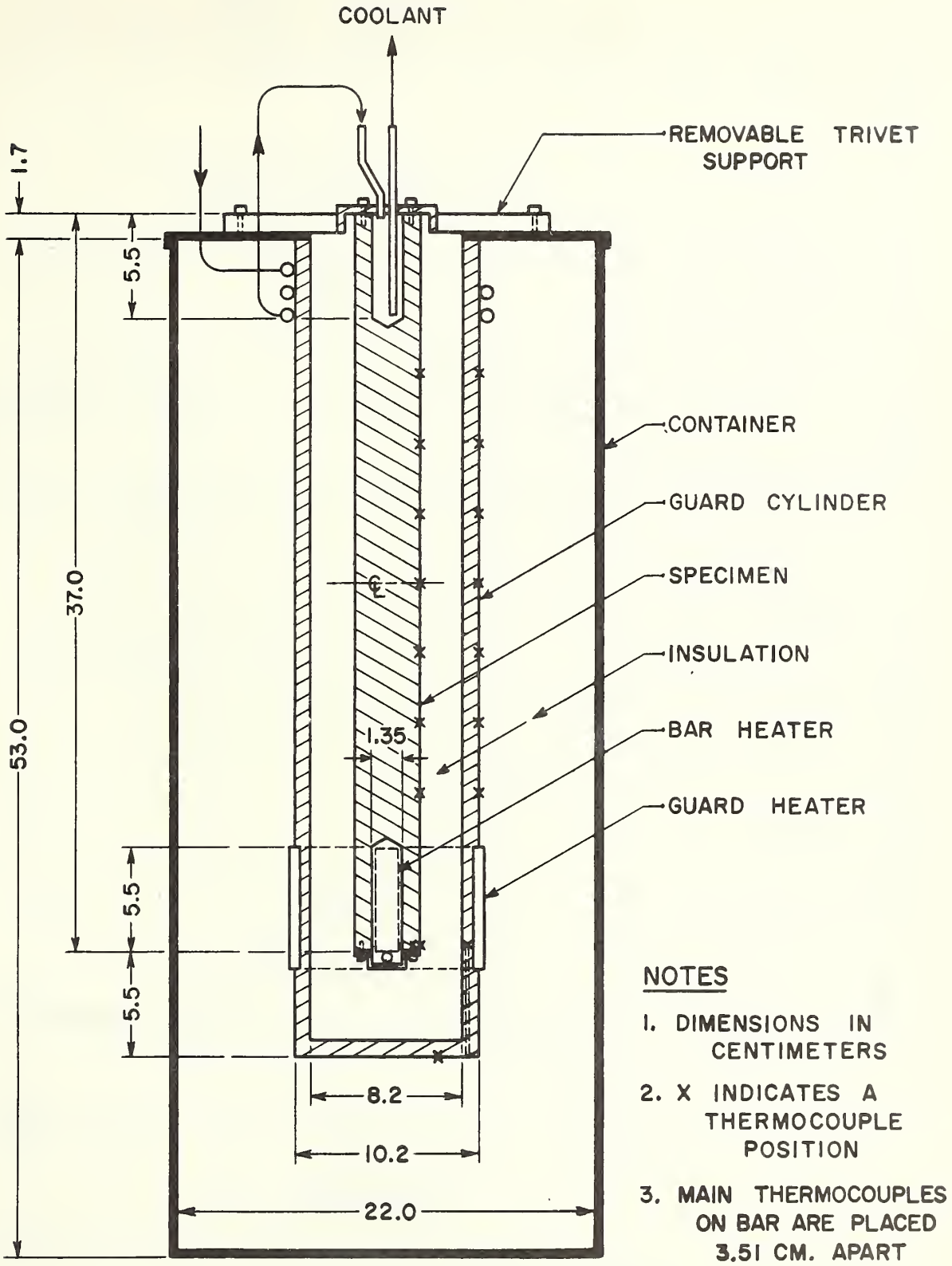
A general qualitative examination did not reveal the presence of other elements.

* By difference

TABLE 2

Thermal Conductivity and Electrical Resistivity
of a
Sample of Electroformed Nickel

Temperature, t °C	Thermal Conductivity, k W/cm deg	Electrical Resistivity, ρ μΩ cm	Lorenz Function $\left(\frac{k\rho}{T}\right)$ V^2/deg^2
− 162	1.078	2.08	2.02×10^{-8}
− 150	1.058	2.42	2.08
− 100	0.981	3.88	2.20
− 50	0.918	5.47	2.25
0	0.863	7.24	2.29
50	0.819	9.24	2.34
100	0.780	11.50	2.41
150	0.744	14.09	2.48
200	0.708	17.05	2.55
250	0.672	20.43	2.63
300	0.636	24.27	2.69
350	(0.600)	(28.64)	2.76
400	0.588	32.34	2.83
450	0.600	34.10	2.83
500	0.611	35.86	2.83
540	0.620	37.26	2.85



APPARATUS FOR MEASURING THE THERMAL CONDUCTIVITY OF METALS

Figure 1

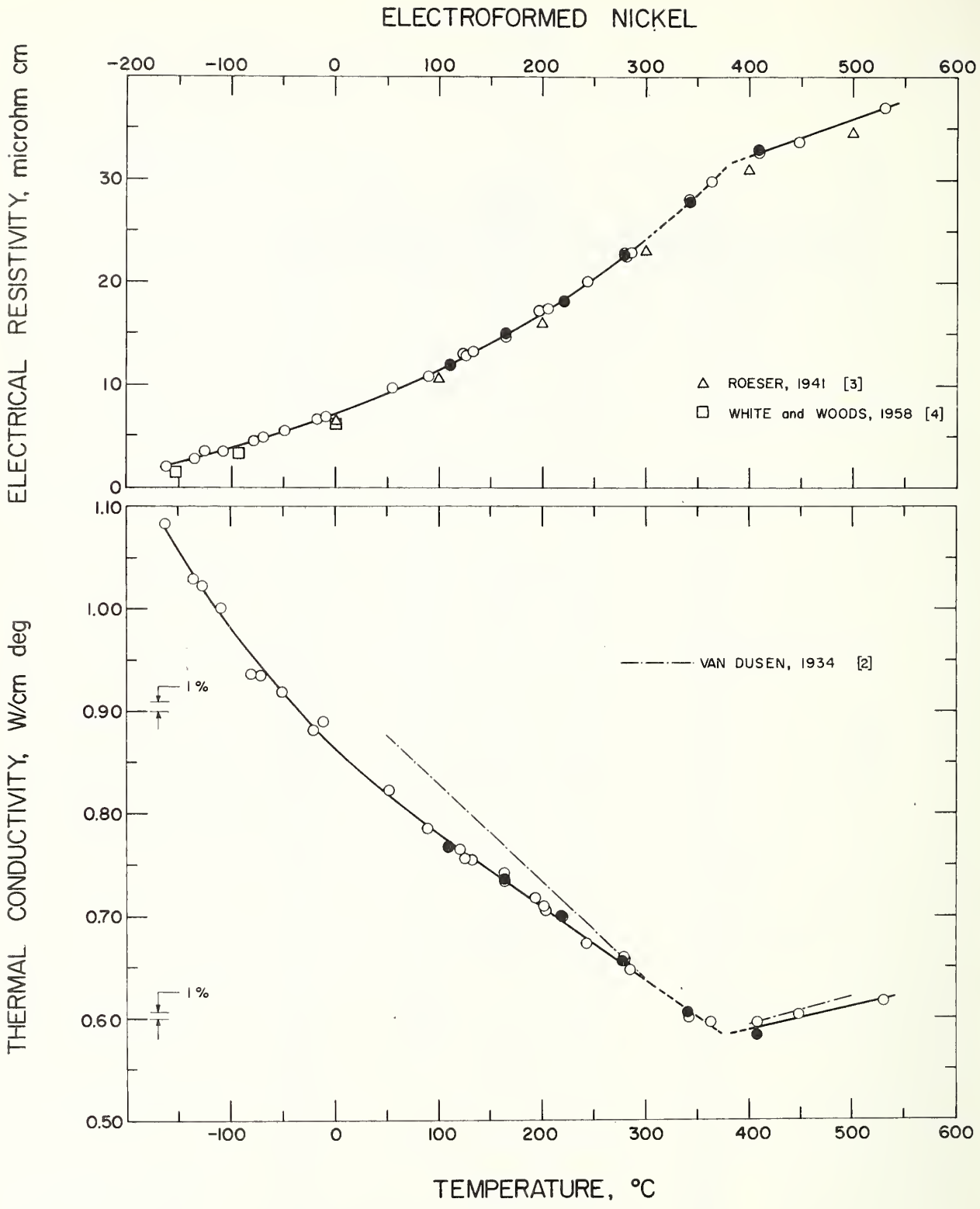


FIGURE 2. THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF ELECTRO-FORMED NICKEL VERSUS TEMPERATURE

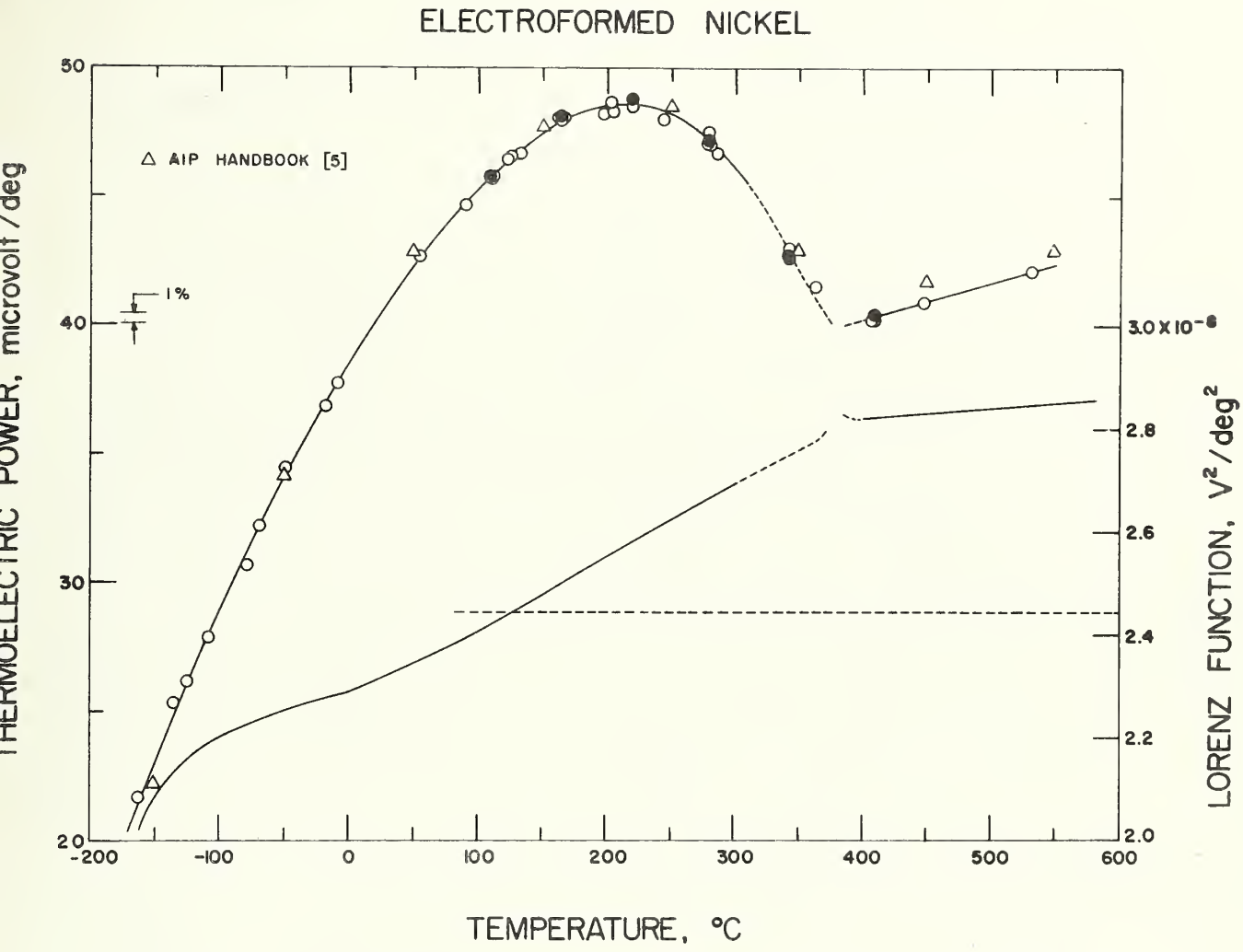


FIGURE 3. THERMOELECTRIC POWER OF ELECTROFORMED NICKEL RELATIVE TO A CHROMEL P THERMOCOUPLE WIRE, AND LORENZ FUNCTION ($k\rho/T$), VERSUS TEMPERATURE

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